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THE USE OF POLYDIALKYL CYCLOSILOXANES AS SOLVENT FOR CHEMICAL  
CLEANING

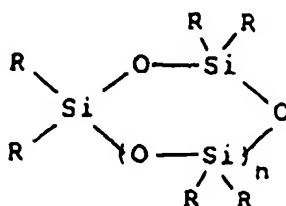
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## [Summary]

Polydialkylcyclosiloxanes of the general formula



in which R is a low-molecular alkyl group with 1 to 4 carbon atoms and n is a whole number from 1 to 3, are used as solvents for chemical cleaning.

### Description

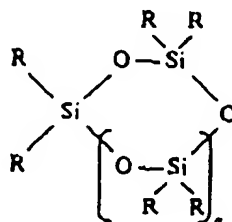
Chemical cleaning is normally carried out with one of the solvents tetrachloroethene (perchloroethylene) or trichlorotrifluoroethane. Since the solvents are environmentally threatening for air and water, chemical cleaning machines are designed so that the solvents that are used are regenerated within the machines by distillation and are recovered from textiles that have come into contact with the solvents in a closed system of evaporation and condensation.

In spite of these precautions one can not always guarantee that small amounts of the halogenated hydrocarbons used as solvents will not escape from the chemical cleaning machines with the cleaned textiles and be breathed in by operating personnel or the customer and pass via the skin into the body. Other problems can arise through defects in storage, transport or handling of halogenated hydrocarbons and because of these unintentionally larger amounts of these solvents can get into the air or into the ground water. Because of the high volatility of these solvents, for example perchloroethylene, there are physiological risks not only for the people who work in chemical cleaning themselves, but also for the vicinity of such operations. Also, there is the danger of contamination of lipophilic foods due to perchloroethylene residues in the air.

The problem has grown in magnitude recently, since it has been established that halogenated hydrocarbons like perchloroethylene are potentially carcinogenic and can damage the liver, kidneys or the nervous system and can paralyze the respiratory center.

The task underlying the invention was thus to find solvents for chemical cleaning that can be used in conventional chemical cleaning processes instead of halogenated hydrocarbons and physiologically are as safe as possible.

In accordance with the invention, as solvents for chemical cleaning one uses polydialkylcyclsiloxanes of the general formula



in which R is a lower molecular alkyl group with 1 to 4 carbon atoms and n is a whole number from 1 to 3.

These polydialkylcyclsiloxanes can be used as substitutes for perchloroethylene and other halogenated hydrocarbons in chemical cleaning, since they have favorable boiling points for the subsequent distillation and are physiologically safe and environmentally friendly. They are free of halogen atoms in the molecule, have a high flash point, in order to avoid the danger of explosions, have a low boiling point, in order to facilitate distillation following use, and they have a density similar to water.

Polydialkylcyclsiloxanes in which R in the above formula means a methyl group are favorable for use in accordance with the invention. In addition, polydialkylcyclsiloxanes in which n is 2, which thus have four silicon atoms in the ring, are especially favorable. Octamethylcyclotetrasiloxane with a density of 0.96, boiling point of 172°C, pour point of 64°C and flash point of 34°C, and decamethylcyclopentasiloxane with a density of 0.96, boiling point of 205°C, pour point of 7°C and flash point of 76°C are, however, also usable.

Surprisingly, the low difference of density with water did not prove to be disadvantageous, but rather to be advantageous in order to increase the cleaning effect. Preferably, the polydialkylcyclsiloxanes of the above formula are used in accordance with the invention in a mixture with 0.1 to 30, especially in a mixture with 5 to 30 wt% water. In addition, it is appropriate to add 0.1 to 5, preferably 1 to 3 wt% of one or more anionic, nonionic or cationic surfactants to the polydialkylcyclsiloxanes.

In the use in accordance with the invention the textiles to be cleaned are fully immersed in the solvent to be used in accordance with the invention or the solvents are made to flow through the textiles, or the textiles are mechanically agitated in them. The solvent that remains uncontaminated by use is then regenerated by distillation and/or filtration or adsorption for reuse.

For regeneration the water content must be separated from the polydialkylcyclsiloxane after distillation, before the latter can be sent for reuse. The usually used gravity separators are not usable because of the low difference of densities with water. For this reason it is expedient to separate the water from the solvent after distillation by means of polyhydric alcohols or polyglycols like glycerol, or in salt solutions. For example, the water-containing distillate of the polydialkylcyclsiloxane is passed through a device containing glycerol, in which the polydialkylcyclsiloxane is fully dehydrated.

If in accordance with the preferred embodiment of the invention a surfactant or surfactant mixture is added, it is a good idea to use a cationic surfactant as cleaning enhancer. Especially preferred as cationic surfactants are quaternary ammonium salts of the type of the difatty alkyl dimethylammonium salts, fatty alkyl benzyl dimethylammonium salts or triammonium salt of citric acid or tricarballic acid. These cleaning enhancers are described in DE Patent 31 11 158 and 31 11 149.

The chemical constitution of these cleaning enhancers which are especially suitable here, can be defined in accordance with the patent Claim 1 of DE Patent 31 11 158. Reference is hereby made to this definition.

The following examples serve to illustrate the invention further. For comparison purposes textiles were treated with water, perchloroethylene, octamethylcyclotetrasiloxane or decamethylcyclopentasiloxane under comparable test conditions. The treated textiles were fabrics and clothing articles of wool, cotton, polyamide, polyester, polyacrylonitrile as well as blends of polyester and cotton, polyester and wool and wool and polyacrylonitrile, that were dyed or undyed. Treatment took place in a laboratory cleaning machine (Wacker drum) with an 8 L drum capacity and 30 cm drum diameter. Treatment conditions were:

#### Example 1

Drum load	400 g textiles
Solvent amount	2000 mL
Drum rotary speed	60 rpm
Treatment time	15 min
Treatment temperature	30°C
Drying (air drying chamber)	70°C
Solvent regeneration, distillation	

To quantify the loss of area (shrinkage) a shrinkage fabric of the International Woolen Secretariat (IWS fabric) was used and the area loss was determined by the change of weight before and after treatment.

The tissues of natural fibers and natural fiber blends treated in water were crinkled and shrunk after treatment.

The fabrics treated in perchloroethylene and in octamethylcyclotetrasiloxane or decamethylcyclopentasiloxane were smooth and visually did not show any change in size. Color loss was not detected. While the substances treated with perchloroethylene felt hard and brittle and the cotton treated in water was stiff and unmanageable, the substances treated in octamethylcyclotetrasiloxane or decamethylcyclopentasiloxane felt pleasantly soft and pliant. The area losses (IWS fabrics) were:

Water	14.6%
Perchloroethylene	1.2%
Octamethylcyclotetrasiloxane	0.8%
Decamethylcyclopentasiloxane	0.9%

Example 2

An artificially soiled test fabric ("Löffler fabric") was treated on the one hand with perchloroethylene and on the other with octamethylcyclotetrasiloxane by the method described in Example 1 and under the conditions listed there. The photometrically measurable brightening of the soil, which consisted of iron oxide and carbon black, was defined as the "cleaning effect." The measurable loss of the degree of whitening of originally white test fabrics that were added to the same test batch was defined as "graying."

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The following were used as cleaning liquid:

Perchloroethylene

Perchloroethylene + 0.5% commercial anionic/nonionic cleaning enhancer (CLIP COMBI)

Octamethylcyclotetrasiloxane

Octamethylcyclotetrasiloxane + 0.5% commercial anionic/nonionic cleaning enhancer (CLIP COMBI)

Octamethylcyclotetrasiloxane + 0.5% CLIP COMBI + 10% water

Result	% Brightening	% Graying
Perchloroethylene	16.5%	5.3%
Per + CLIP COMBI	24.8%	1.1%
Octamethylcyclotetrasiloxane	14.6%	5.9%
Octamethylcyclotetrasiloxane + CLIP COMBI	18.4%	4.6%
Octamethylcyclotetrasiloxane + CLIP COMBI + 10% water	47.7%	2.7%

Although pure octamethylcyclotetrasiloxane has a somewhat lower cleaning effect than perchloroethylene, a considerable increase of effect develops when water is added.

Example 3

To determine the distillation behavior and distillation stability in the presence of water, which can be present either as conditioner moisture from natural fiber textiles or as an addition to improve the cleaning effect during distillation, octamethylcyclotetrasiloxane or hexamethylcyclotetrasiloxane were each mixed with 1% anionic or 1% cationic commercial cleaning enhancer and 10% water in a distillation apparatus. The mixtures were distilled discontinuously (Soxhlet) for 40 h, in a circulation system, by returning the distillate to the distillation receiver.

The change of the pH of the distilled water as well as the refractive index change of the cyclotetrasiloxanes were evaluated.

No change of the refractive index had occurred after 40 h of distillation, but the pH of the distilled water showed a change from 7.1 to 6.2 in the sample in which cationic cleaning enhancer was used.

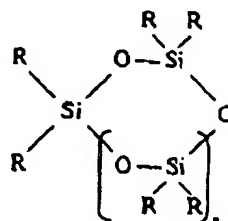
A second test sought to determine how the distilled water can be separated simply from the cyclosiloxane, since with normal distillation a coarse dispersion that remains stable for a relative long time is formed and takes a long time to separate because of the low difference of densities.

For this reason the distillate was sent to a high density hygroscopic liquid (glycerol or magnesium chloride solution), in which the water was removed by absorption, so that the distillate was clear and water-free.

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### Claims

1. The use of polydialkylcyclosiloxanes of the general formula



in which R is a lower molecular alkyl group with 1 to 4 carbon atoms and n is a whole number from 1 to 3, as solvent for chemical cleaning.

2. A use of polydialkylcyclosiloxanes as in Claim 1, where R means the methyl group.
3. A use of polydialkylcyclosiloxanes as in Claim 1 or 2, where n means 2.
4. A use of polydialkylcyclosiloxanes with a water content from 0.1 to 30, preferably 5 to 30 wt%, as in one of Claims 1 to 3.
5. A use of polydialkylcyclosiloxanes with a content of 0.1 to 5, preferably 1 to 3 wt%, of at least one anionic, nonionic and/or cationic surfactant, according to one of Claims 1 to 4.
6. The use of polydialkylcyclosiloxanes as in Claim 4 or 5, with distillation of the used polydialkylcyclosiloxanes and subsequent separation of the water by absorption on at least one polyhydric alcohol or polyglycol or in a salt solution and return of the thus purified polydialkylcyclosiloxanes for reuse.

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